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CERTIFICATE

This certificate is issued in support of an application for Patent registration in a country outside New Zealand pursuant to the Patents Act 1953 and the Regulations thereunder.

I hereby certify that annexed is a true copy of the Provisional Specification as filed on 19 November 2003 with an application for Letters Patent number 529640 made by MATTERSMITHS HOLDINGS LIMITED.

Dated 13 December 2004.



Neville Harris
Commissioner of Patents, Trade Marks and Designs



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529640

Patents Form No. 4

Our Ref: SHR504620

Patents Act 1953
PROVISIONAL SPECIFICATION
IMPROVED TREATMENT PROCESS

We, **MATTERSMITHS HOLDINGS LIMITED**, a New Zealand company, of 35
Renoir Street, West Harbour, Auckland New Zealand do hereby declare this
invention to be described in the following statement:

PT043861650

Intellectual Property
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IMPROVED TREATMENT PROCESS

FIELD

5 This invention relates to methods of conditioning organic substrates. In particular, the invention relates to methods of conditioning lignocellulosic substrates. The conditioning may be for the purpose of facilitating subsequent impregnation of the substrate with preservatives or like compositions.

BACKGROUND

10 Lignocellulosic material, and more specifically debarked logs or sawn lumber, largely because these are of biological origin, are prone to attack by organisms such as bacteria, insects, nematodes and a variety of fungi including decay and staining fungi. Such attack reduces the service life of logs or lumber extracted there from, degrades the appearance of the logs or lumber, and reduces the service life of such materials with resultant cost of replacement or potential hazard due to failure.

15 Logs, or lumber, when freshly cut, generally are not contaminated internally by fungi or insects. However, subsequent to felling these become vulnerable to attack by insects and fungi, particularly decay fungi. These can degrade these substrates relatively quickly depending on the species concerned.

20 To mitigate degradation by biological pests, methods have been developed to treat these substrates with a variety of chemicals by various physical processes.

25 Whilst it is possible to impregnate lignocellulosic material such as lumber products with preservatives and like compositions, by a number of variations of vacuum and pressure cycles, these are generally restricted to treatment of the dry substrate because free space must be available within the substrate to receive the composition. This necessitates prior drying and this can be time consuming and/or expensive. Similarly such processes necessarily include injection of large volumes of solvent and which must later be removed by a drying process. Use of such solvents also necessitates storage of large volumes of potentially toxic fluid.

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35 Another method, for use with green substrates (thus avoiding the necessity for drying),

utilises a process wherein the green substrate is conditioned in a steam autoclave and, after a subsequent period of cooling and moisture equilibration, the substrate is treated by alternating pressure and vacuum cycles. In this steam conditioning process, green substrates are treated with heat directly using a condensing medium, such as live steam, 5 in an autoclave where the pressure can be maintained at up to 30 pounds per square inch over pressure. The substrate is heated to the required temperature, preferably above 100 Celsius, at elevated pressure. Upon reaching the required temperature the autoclave is vented allowing the pressure to drop rapidly and thus allowing the water to boil and the moisture to escape as steam.

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During the early part of this process the steam may be present as a vapour or condensed as a liquid phase. The latter is not preferred because it is highly contaminated by wood extractives. In the later part of the process the water present is entirely in the gaseous state.

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In this steam conditioning process radial cells in the substrate material, known as ray cells or parenchyma, are also emptied by the internal pressure. This then provides an avenue through which the preservative solution can enter when subsequently applied:

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There are several disadvantages to this steam conditioning process for treating green substrates; (a) because the substrate begins at ambient temperatures, there is substantial condensation of the steam on the substrate and this becomes contaminated with wood extractives. The volume of condensate becomes significant, is highly contaminated, and is expensive to dispose of; (b) the initial part of the process is maintained at atmospheric pressure to remove any air from the chamber. This is because air is a poor conductor of heat and would otherwise reduce the rate at which the substrate can be heated. This leads to a costly wastage of steam; (c) the heating process is slow because it relies entirely on heat conduction from outside the substrate to the centre of the substrate; and (d) when heated for long periods at temperatures of 130°C which is typical of such

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processes, the substrate loses significant physical strength. This lengthy heating step thus degrades the physical properties of the substrate. This effect is exacerbated because to get energy flow to the centre of the substrate requires a high temperature gradient. Thus the surface of the substrate may be at 130 Celsius whereas the centre may have only reached 100 Celsius at the end of the process. Since strength degradation 30 is significantly greater at 130 Celsius than at 100 Celsius, the outer parts of the substrate thus degrading whilst the centre is coming up to temperature. For example, large diameter

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poles may be heated for up to 15 hours with the temperature external to the pole being at 130 Celsius. This can significantly degrade the strength of the pole.

US Patent 6596975 to Vinden and co-workers teaches a method of increasing the permeability of wood substrates using microwave energy with frequency of 0.1 to 24GHz. It teaches a method for increasing the permeability of wood which comprises subjecting wood with moisture content (based on dry weight) of at least 15% to microwave radiation at a frequency in the range of from about 0.1 to about 24 GHz. It is our contention that microwaves can be used for small pieces only because penetration is directly proportional to wave length. Microwaves at a commercial frequency of 2.45 GHz for example have a wave length of 0.12 metres and at 24 GHZ the wavelength is 1.25 centimetres and is therefore unsuitable for large chambers or large volumes of wood.

The process of Vinden provides no pressure constraint therefore as the temperature rises the pressure will be continuously venting and therefore the bulk of the substrate can not be maintained at any time at a temperature of 100 Celsius or greater.

Radio frequency heating at a commercial frequency of 27 MHz which has a penetration of several metres has been used to dry lumber. This can be significant when handling large pieces or packets or bundles of wood. At 27 to 40 MHz the wave length is in the order of 7 to 11 metres. This is easily adequate to penetrate large chambers that can hold large pieces or packets or bundles of wood, for example, and the wood they contain. The use of low frequency RF for drying requires continuous venting of moisture vapour and thus there is no pressure build up.

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OBJECT OF THE INVENTION

It is an object of the invention to provide an alternative process for conditioning organic substrates, such that the substrate is amenable to concurrent or subsequent treatment, that overcomes or ameliorates at least some of the prior art problems. It is an alternative object to provide the public with a useful choice.

STATEMENT OF THE INVENTION

In broad terms, the invention includes a method of conditioning an organic substrate; the method including the steps of:

- (a) Subjecting the substrate to low frequency RF energy in a constrained environment for a time sufficient to heat at least part of the moisture contained in the substrate to a temperature of or above 100°C;
- (b) Releasing the pressure created in the constrained environment in a manner causing the moisture within the substrate to boil or evaporate.

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Preferably the low frequency RF energy is between about 10 and 100 MHz; more preferably between about 27 and 40 MHz.

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Preferably the temperature achieved within the substrate is between 100 and 130 Celsius.

Preferably the substrate is a lignocellulosic material.

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Preferably the lignocellulosic material has a moisture content of more than 60% based on dry weight of the material; preferably greater than 100%.

Preferably the moisture content may be less than 30% based on dry weight of material.

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Preferably the substrate is then stored to allow the temperature and moisture in the substrate to equilibrate.

Preferably the substrate is concurrently or subsequently impregnated with a composition.

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Preferably in one aspect the composition is an aqueous solution that preferably contains polar and/or non polar solvents; pesticidal or preservative components; and/or polymeric or pre-polymeric components.

Preferably in one aspect the composition is a volatile pesticidal or preservative component; and/or pre-polymeric component.

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In another embodiment, the invention may be seen to comprise an organic substrate, preferably a lignocellulosic substrate that has been conditioned according to the method of the invention, preferably the substrate will have been concurrently or subsequently impregnated with a composition as well.

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DESCRIPTION OF THE INVENTION

The following is a description of the preferred forms of the invention given in general terms in relation to the application of the present invention. While the description focuses 5 particularly on the delivery of composition in lumber or logs, it should be appreciated that the method may be applicable to other substrates.

In general terms, the invention relates to methods of conditioning an organic substrate, and, preferably a lignocellulosic substrate, using RF energy in the low frequency range 10 (between about 10 and about 100 MHz). During this process the pressure is constrained such that an over pressure is applied to the substrate. Since water boils at greater than 100 Celsius when the pressure is greater than atmospheric, the water within the substrate can reach temperatures higher than 100 Celsius without boiling. When the pressure is quickly released (e.g. by venting), the water in the substrate will be above its boiling point 15 and therefore will immediately boil and convert to steam. This creates substantial pressure within the substrate which forces remaining water and cellular debris to be ejected from the substrate. This creates voids within the substrate and also clears pathways into the substrate sufficient for subsequent treatment with desired compositions.

20 The conditioning method of the invention may be used to condition a substrate for subsequent delivery of a liquid composition. The composition will preferably be an aqueous solution and may contain polar and/or non polar solvents and the like for example alcohol and vegetable oils. If the composition is to be applied to the substrate when the substrate is at temperature higher than ambient then the components of the 25 composition will preferably be non-volatile at the temperature of the substrate at the time of application of the composition. Persons generally skilled in the art to which the invention relates will no doubt appreciate various compositions that may be applicable in the process of the invention. However, by way of example, where treatment or prevention of infection or pre-infection by pest organisms is desired, compositions (biocide 30 compositions) having pesticidal (fungicidal, bactericidal, insecticidal for example) or preservative properties may be used. Where it is desired that the substrate has increased density or strength properties, compositions containing polymeric or pre-polymeric components may be useful. Similarly compositions may include those of use in waterproofing a substrate. Additionally compositions containing certain dyes to colour the 35 substrate may be used.

To this extent the invention in one aspect includes the coupling of the conditioning step with the step of subsequently impregnating the substrate with a desired composition.

The conditioning method of the invention may also be used to condition a substrate for concurrent delivery of a composition. The composition will preferably include a biocidal component and/or a pre-polymeric component. If the composition is to be applied to the substrate when the substrate is at temperature higher than ambient then the components of the composition may be volatile at the temperature of the substrate at the time of application of the composition. Persons generally skilled in the art to which the invention relates will no doubt appreciate various compositions that may be applicable in the process of the invention. However, by way of example, where treatment or prevention of infection or pre-infection by pest organisms is desired, compositions (biocide compositions) having pesticidal (fungicidal, bactericidal, insecticidal for example) or preservative properties may be used. Where it is desired that the substrate has increased density or strength properties, compositions containing polymeric or pre-polymeric components may be useful.

As used herein, "organic substrate" should be taken to mean any organic material which may be in need of delivery of a composition of some nature; for example for the purposes of protection or treatment to prevent or ameliorate growth of pest organisms. Such substrate is preferably lignocellulosic, for example, wood products, lumber or logs. The only requirement is that there is sufficient moisture capable of being evaporated by the energy imparted by the RF energy used subsequent to the pressure constraint being removed. While single pieces of lumber could be treated by the inventive process, it is envisaged that treatment of a number of pieces consolidated into a packet or bunch, perhaps 200 pieces or more, would be treated by the present process. These might comprise sawn lumber, poles or logs for example.

In the case of lignocellulosic substrates, those which "contain a level of moisture" include freshly felled and debarked logs or freshly sawn lumber (so called green lumber). Those of general skill in the art to which the invention relates will be aware that freshly felled logs or freshly sawn lumber will contain approximately 150% of their dry weight as moisture if soft wood and approximately 80% if hard wood.

"Pests" or "pest organisms", as referred to herein may include any organisms which may infect an organic substrate such as wood. While the invention is particularly applicable to

fungi pest organisms may also include bacteria insects, nematodes and the like.

When used herein the term "treatment" should be taken in their broadest possible context. It should not be taken to imply that a substrate has treated such that pest organisms are 5 totally removed, although this would obviously be preferable. Prevention and amelioration of growth of pest organisms is also encompassed by the invention.

It is therefore one preferred form of the invention to utilise low frequency RF energy to heat a substrate, preferably a green substrate, in a constrained environment followed by 10 rapid release of the pressure created at a suitable time, transfer of that substrate to dry storage, and then transfer of the substrate to a pressure vessel where impregnation with preservative or like composition can be undertaken using variations of vacuum pressure cycles if desired.

15 In a preferred embodiment, the conditioning method will include the steps of:

- (a) subjecting a substrate (and preferably a green substrate) to low frequency RF energy in a constrained environment for a time sufficient to heat moisture contained in the substrate sufficient to raise the temperature within the substrate to 100 Celsius or more; and
- 20 (b) releasing the pressure to allow the moisture within the substrate to boil and/or evaporate

In another preferred embodiment, the conditioning method will include the steps of:

- (a) subjecting a substrate to low frequency RF energy in a constrained environment for a time sufficient to heat moisture contained in the substrate sufficient to raise the temperature within the substrate to 100 Celsius or more; and
- 25 (b) incorporating into the void surrounding the substrate in the constrained environment, a composition which may impart sterilisation, preservative, or property modifying aspects;
- 30 (c) releasing the pressure to allow the moisture within the substrate to boil and/or evaporate

The temperature and moisture can then be allowed to equilibrate prior to proceeding with another treatment process, such as impregnation with a composition, or machining and 35 the like.

The period of time the substrate is subjected to the low frequency RF energy is a time sufficient to heat the substrate uniformly and create a temperature of 100 Celsius or greater within the substrate. The temperature created should be one which is sufficient to elevate moisture temperature within the substrate to at least 100 Celsius.

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It should be appreciated that temperatures and times sufficient to heat the substrate may vary depending upon factors such as the nature of the substrate, the frequency of the RF energy, and the temperature ultimately required. The phrase "sufficient to heat" does not imply that the conditioning process must be sufficient to completely rid the substrate of moisture. The inventor contemplates a reduction in any moisture and enhancement of penetrative pathways being appropriate in respect of the invention.

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A constrained environment is any 'chamber' which may be sealed against pressure drop during the process of the invention or which may have sufficient supplementary pressure that the boiling point of water within the substrate is raised above 100 Celsius. Typically such a chamber is cylindrical for obvious physical reasons (a cylinder can not have any sides which could expand) and has appropriately domed doors. Such environments will be well known to a skilled person in this field. Typically suitable autoclaves can be used. If the process of the invention operates at a lower temperature (close to 100 Celsius) the over pressure might only be a few psi, in which case almost any chamber could be used as long as the pressure could be constrained within the chamber.

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Venting, or release of pressure, would be achieved as for current practice. This is typically a series of large diameter gate valves. These are abruptly and simultaneously opened to maximise pressure drop and thus enhance the boiling effect.

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In a particular embodiment, a substrate with relatively high moisture content, for example green lumber with a moisture content of greater than 100% (approximately 150%) based on dry weight, would be heated using the low frequency RF energy whilst under constraining pressure. In such a case the heating would heat the moisture within the substrate sufficiently to exceed the boiling point of water. Upon rapid venting of pressure this would enable the water to boil and escape the substrate as steam resulting in the creation of voids in the substrate. Subsequently, if the temperature of the substrate is at or about ambient then vacuum pressure techniques may be used to impregnate the substrate preferably with a liquid composition. This impregnation can be assisted by use of vacuum/high pressure techniques particularly when the temperature differential is not

sufficient to ensure adequate impregnation without assistance. It would be an advantage if the composition itself had properties which allowed it to penetrate the substrate to some extent (as opposed to just sitting on its surface following application).

5 Of course, when heating under a pressure constraint (e.g. in a suitable autoclave), the temperature created within the substrate can considerably exceed 100°C while the boiling of the water contained within the substrate will not take place. At a time and temperature decided upon, the pressure constraint can be removed thus allowing the moisture to boil initially at a temperature substantially above 100°C.

10 The substrate is heated using radio frequency energy preferably at frequencies of less than 100 MHz. More specifically, frequencies of approximately 27 to 40 MHz are particularly desirable. At this range of wavelengths, batches of two or more separate pieces of a substrate, for example two or more logs of lumber may be treated simultaneously. In a commercial operation this may involve treatment of perhaps 30 cubic metres of logs or lumber. The invention is preferably applied to batch processing. As low frequency RF is used (for example 27 to 40 MHz) a number of logs may be sufficiently and effectively heated simultaneously. Whilst packets of lumber may be 2 or 3 metres in length some may be longer (for example, poles may be 10 metres long or more). To 15 ensure penetration throughout such a packet, long wavelength energy must be used otherwise it will be absorbed only superficially. For example, RF energy at 27 MHz has a wavelength of over 11 metres thus the energy will be distributed adequately throughout the substrate ensuring that moisture in the substrate is mobilised as discussed earlier.

20 Whereas prior art may take up to 15 hours to adequately precondition the substrate, because heating relies entirely on conduction, the process of this invention can achieve the same target temperature within 3 hours or less depending on the energy available. Because the heating is throughout the substrate during the process the only constraint on 25 time is the energy available from the RF source.

30 Several processes are available to impregnate substrates using vacuum/pressure systems. Such vacuum and pressure cycle variations include Reuping, Lowry and Full Cell processes. These processes are adequately described in "Industrial Timber Preservation", 1979, J G Wilkinson, Associated Business Press. Any process to 35 impregnate a substrate preconditioned by the method of the present invention could be

used as desired. The method of the invention facilitates the impregnation of such substrates as is discussed earlier herein.

5 The invention further extends to organic substrates that have been preconditioned according to the process of the invention and also to organic substrates that have been preconditioned by the process of the invention and then impregnated with a composition as desired.

10 **EXAMPLES**

The invention will now be further described with reference to the following non-limiting examples.

15 **Method - green substrate**

We envisage one or more variations of a process wherein the substrate is constrained in the chamber and an over pressure applied and the substrate heated using low frequency RF energy. Two options include an over pressure supplied from an external source and the other from over pressure created within the process itself.

20 **Example 1**

Green lumber is placed in an autoclave and the doors closed to create a constrained environment. An over pressure within the autoclave of 30 pounds per square inch is applied from an external source. This can be any gas and is most economically air.

25 Energy is then applied in the form of radio frequency energy preferably at a frequency below 100 MHz and more preferably at 27 or 40 MHz, thus immediately heating the substrate, and without need for venting or slow heat up. No condensate is created in this process because no live steam is applied and because any water within the substrate is constrained. After a time when sufficient energy has been applied to achieve the desired 30 temperature throughout the substrate, typically between 100 and 130 Celsius, the pressure is rapidly released (the autoclave is rapidly vented) whereupon water within the substrate will boil causing evaporative loss and clearing the substrate of cellular debris as occurs for the traditional steaming process.

35 In another aspect this process might include addition, in the gaseous phase, of volatile biocide components which may then impart a sterilising or preserving action to the

substrate and/or pre-polymeric components which might then impart a change to the physical properties of the substrate. For example, it is known to those versed in the art that biocides such as orthophenylphenol or the like have a relatively high vapour pressure which increases with temperature. Thus inclusion of this art in this invention can impart 5 beneficial properties to the substrate. Similarly boric acid is volatile in the presence of gaseous water and could thus be presented to the substrate by the process of this invention thus eliminating the need for a liquid or vapour solvent phase. Further it is also known that the rate of diffusion of such biocides within the substrate is significantly enhanced as temperature rises. Thus once incorporated in or on the surface of the 10 substrate the elevated temperature will facilitate diffusion into the interior of the substrate. In particular where the biocide is conducted within the liquid phase within the substrate it can be important to maintain the liquid phase by an over pressure during this process. This can be otherwise unachievable due to evaporative loss if the over pressure is not 15 present. It should be appreciated that the use of steam to carry biocidal components (or the use of other volatile components) is not part of the heating step, but is rather a means of providing the biocidal components to the substrate.

The substrate is then moved to temporary storage to allow cooling or moisture equilibration.

20 Following storage, the lumber is again transferred to allow application of the preservative composition of choice. The composition can be applied using known techniques and impregnation can be assisted or achieved using known vacuum/high pressure techniques as available.

25 **Example 2**
Green lumber is placed in an autoclave which is then hermetically sealed to create a constrained environment. No initial overpressure is applied, but as energy is then applied in the form of radio frequency energy preferably at a frequency below 100 MHz and more 30 preferably at 27 or 40 MHz, an overpressure develops in the autoclave from steam generated by direct heating of the substrate and thermal expansion of air in the autoclave and the wood by indirect heating. Some condensate is created during pressure development but is minimal compared to conventional steaming or kiln conditioning and will revert to a gas as the temperature within the autoclave rises.

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As described in example 1 biocides, pre-polymers and other components can be included in the gaseous or vapour phase enclosing the substrate.

5 After a time when sufficient energy has been applied to achieve the desired temperature throughout the substrate, typically 100 to 130 Celsius, the pressure is rapidly released whereupon water within the substrate will boil causing evaporative loss and clearing the substrate of certain cellular debris as occurs for the traditional steaming process.

10 The substrate can be moved to temporary storage and treated as referred to in Example 1.

Further Examples

15 Further examples would be as for examples 1 and 2 but wherein the moisture content of the substrate is lower, typically below 30 per cent, in which case the additional compositions such as biocides or pre-polymeric components may in part diffuse into the substrate in a gaseous phase due to the lower substrate moisture content.

The benefits of the process of the invention can be any one or more of;

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- Significant reduction of liquid waste streams and the cost of disposal thereof because there is no condensate from steam.
- Elimination of the pre-heat air flushing period thus saving significant time and energy cost.
- A significant reduction in heating time because energy is transferred directly throughout the substrate.
- A reduction in strength loss because there is a significant reduction of time at the elevated temperature.
- A reduction in strength loss because the substrate may be processed at lower average temperature.

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- Lower energy consumption because less energy is wasted by elimination of the heat up period and more efficient energy transfer.
- Elimination of energy required for heating the metal autoclave and bogies for carrying the substrate because these are inert to RF energy.

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- A significant reduction in processing time because the rate of heating does not depend on conduction.

5 Overall the process is significantly simplified, energy costs are lowered and waste streams substantially reduced. The total period for the process is entirely related to the rate of energy input not to the conductivity of the substrate. Therefore if sufficient RF energy is available the process time can be significantly shortened.

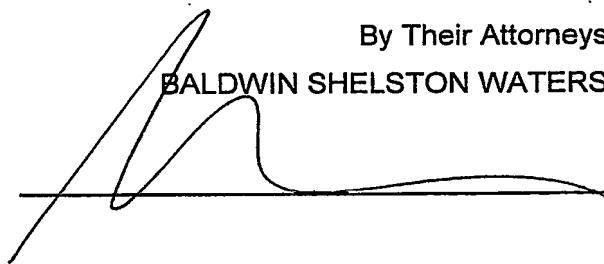
10 Thus we envisage a fast energy efficient process and which eliminates a number of problems and costs normally associated with preservation of logs, poles or lumber.

15 The foregoing includes a disclosure of the invention including preferred forms thereof. Alterations or modifications that would be readily apparent to the skilled person are intended to be included within the scope of the invention. References to prior art herein, unless otherwise stated, does not constitute an admission that such art constitutes common general knowledge in New Zealand or any other country.

20 MATTERSMITHS HOLDINGS LIMITED

By Their Attorneys

BALDWIN SHELSTON WATERS



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